weakly and the data is relatively poor). Final $R1 = 0.096$ $[I > 2\sigma(I)]$ and $wR2 = 0.265$ (all data).[14] All four of the C atoms of the THF molecule bonded to Li(5) are disordered over 50:50 sites. The H atom attached to N(4), which is inferred from the IR spectrum of **1**, was located in the final difference map. Crystallographic data (excluding structure factors) for the structure reported in this paper have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication no. CCDC-144824. Copies of the data can be obtained free of charge on application to CCDC, 12 Union Road, Cambridge CB21EZ, UK (fax: (+44)1223-336-033; e-mail: deposit@ccdc.cam.ac.uk).

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Stabilization of a Pentastibacyclopentadienyl Ligand in the Triple-Decker Sandwich Complexes $[\{(\eta^5\text{-}1,2,4\text{-}t\mathrm{Bu}_3\mathrm{C}_5\mathrm{H}_2)\mathrm{Mo}\}_2(\mu,\eta^5\text{-}\mathrm{Sb}_5)]$ and $[(\eta^5\text{-}1,2,4\text{-}t\mathrm{Bu}_3\mathrm{C}_5\mathrm{H}_2)\mathrm{Mo}(\mu,\eta^5\text{-}\mathrm{Sb}_5)\mathrm{Mo}(\eta^5\text{-}1,4\text{-}t\mathrm{Bu}_2\text{-}2\text{-}\mathrm{MeC}_5\mathrm{H}_2)]$**

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Triple-decker sandwich complexes containing *cyclo*-(η^n-E_n) ligands ($n = 5, 6$) are well known for E = P, As.[1–3] Although elemental pnictogens (white phosphorus, P_4, and yellow arsenic, As_4) are the most obvious reagents and have been used in many cases, organophosphorus[4] and organoarsenic[1] rings, and $[(CO)_5CrPCl_3]$[5] are also viable sources of phosphorus and arsenic elemental ligands. Since an Sb_4 molecule is not available for synthetic purposes, other starting materials have been used for the preparation of complexes with substituent-free antimony ligands: both metallic antimony[6a] and *cyclo*-$Cp^*_4Sb_4$[6b] ($Cp^* = Me_5C_5$) gave complexes containing Sb_2 fragments. *cyclo*-$t\mathrm{Bu}_4\mathrm{Sb}_4$[7] proved to be a versatile reagent in the synthesis of complexes containing both Sb_2 and *cyclo*-Sb_3 ligands.[8] Compounds containing *cyclo*-Sb_5 ligands were observed as decomposition products in the mass spectra of clusters containing Sb_2 and Sb_3 fragments but have not yet been identified in the condensed phase.[8b] We report here on the stabilization of the pentastibacyclopentadienyl ligand *cyclo*-(η^5-Sb_5) as the middle-deck in the triple-decker sandwich complexes **1** and **2**.

$[(\eta^5\text{-}1,2,4\text{-}t\mathrm{Bu}_3\mathrm{C}_5\mathrm{H}_2)\mathrm{Mo}(\mu,\eta^5\text{-}\mathrm{Sb}_5)\mathrm{Mo}(\eta^5\text{-}1,2,4\text{-}t\mathrm{Bu}_3\mathrm{C}_5\mathrm{H}_2)]$ **1**

$[(\eta^5\text{-}1,2,4\text{-}t\mathrm{Bu}_3\mathrm{C}_5\mathrm{H}_2)\mathrm{Mo}(\mu,\eta^5\text{-}\mathrm{Sb}_5)\mathrm{Mo}(\eta^5\text{-}1,4\text{-}t\mathrm{Bu}_2\text{-}2\text{-}\mathrm{MeC}_5\mathrm{H}_2)]$ **2**

Complexes **1** and **2** are formed when a mixture of *cyclo*-$t\mathrm{Bu}_4\mathrm{Sb}_4$ and $[\mathrm{Cp'''(CO)_3MoCH_3}]$ ($\mathrm{Cp'''} = \eta^5\text{-}1,2,4\text{-}t\mathrm{Bu}_3\mathrm{C}_5\mathrm{H}_2$)[9] is heated in decaline. It is not surprising that a by-product of the thermal reaction is $[\{\mathrm{Cp'''Mo(CO)_2}\}_2]$ (*Mo*≡*Mo*) (**3**). Unexpected, however, is the formation of the complex **2** by replacement of one of the *tert*-butyl groups in position 1 or 2 on one of the cyclopentadienyl rings in the compound **1** by a methyl group. The triple-decker sandwich complexes **1** and **2** could not be separated by chromatography. After chromatographic separation from the reaction mixture an air-sensitive, dark green, microcrystalline solid containing both **1** and **2** was obtained in about 18% overall yield. The two complexes are very soluble in hydrocarbons, giving emerald-green solutions that are air-sensitive. These solutions immediately change color to brown when exposed to air. A few relatively large single crystals of **2** were grown from a solution of the mixture in hexane. The identity of the compounds was confirmed by elemental analysis and by ^{1}H NMR and IR (**3**) spectroscopy and mass spectrometry. The mass spectra suggested a mole ratio of 4:1 (**1**:**2**) in the mixture. The complexes **1** and **2** have a remarkable thermal stability, the mixture melting without significant decomposition at 331–333 °C and subliming at 260 °C/10^{-3} Torr.

The crystal structure of **2** was determined by X-ray crystallography.[10] The molecule has a typical triple-decker sandwich structure (Figure 1), with the bulky cyclopentadienyl rings almost parallel to each other (dihedral angle 5.3°) and to the Sb_5 ring (3.2° each). The Sb_5 middle deck is very slightly folded towards Mo(2), with Sb(2) lying 20 pm out of the plane formed by the other four antimony atoms. The Sb–Sb bonds (275.59(11) and 276.56(9) pm) involving Sb(2) are shorter than the single bonds in *cyclo*-$t\mathrm{Bu}_4\mathrm{Sb}_4$ (Sb–Sb 281.7(2)–282.1(2) pm)[11a] or *cyclo*-$((\mathrm{Me_3Si})_2\mathrm{CH})_4\mathrm{Sb}_4$ (282.1(1)–287.8(1) pm),[11b] and similar to those in the clusters $[\mathrm{Cp(CO)_2MoSb_3}]$ (273.45(10)–278.05(11) pm) and $[\mathrm{Cp^*(CO)_2MoSb_3}]$ (273.97(9)–276.82(8) pm).[8a] They are considerably longer than an Sb–Sb double bond (264.2(1) pm in $\{2,4,6\text{-}[(\mathrm{Me_3Si})_2\mathrm{CH}]_3\mathrm{C_6H_2}\}_2\mathrm{Sb}_2$).[12] The Sb–Sb bonds involving Sb(5) are longer (284.24(9) and 285.04(9) pm), their lengths falling in the normal range for Sb–Sb single bonds. The Sb(3)–Sb(4) bond length has an intermediate value (279.78(8) pm). A

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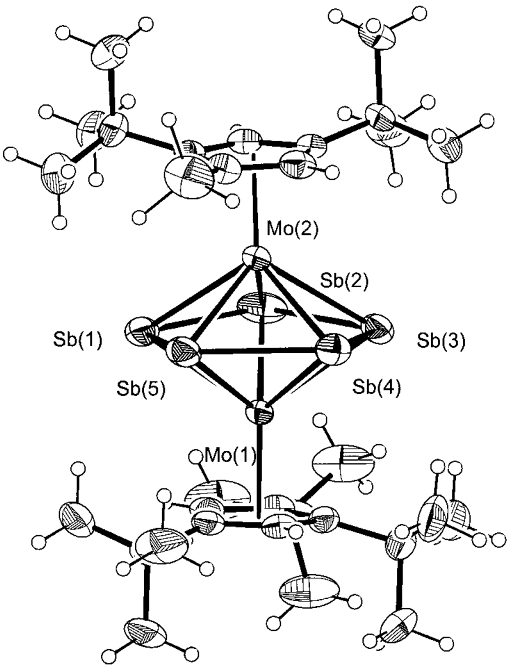

Figure 1. Molecular structure of **2** in the crystal. Selected bond lengths [pm] and angles [°]: Sb(1)-Sb(2) 275.59(11), Sb(2)-Sb(3) 276.56(9), Sb(3)-Sb(4) 279.78(8), Sb(4)-Sb(5) 284.24(9), Sb(1)-Sb(5) 285.04(9), Mo(1)-Mo(2) 296.40(8), Mo(1)-Sb 277.40(9) – 290.23(8), Mo(2)-Sb 272.76(8) – 283.92(9), Mo-$Cp_{centroid}$ 201.1 (av); Sb(1)-Sb(2)-Sb(3) 108.06(3), Sb(2)-Sb(3)-Sb(4) 110.13(3), Sb(3)-Sb(4)-Sb(5) 105.82(2), Sb(4)-Sb(5)-Sb(1) 107.63(3), Sb(5)-Sb(1)-Sb(2) 107.86(2), Mo(1)-Sb-Mo(2) 62.43(2) – 65.19(2), Sb-Sb-Mo 57.40(2) – 63.28(2).

similar but folded five-membered ring is present in an Sb_5^{5-} Zintl ion, whose Sb–Sb bonds are on average 5 pm longer than those in **2**.[13]

Although the Sb_5 ring in **2** is not as regular as the pnictogen rings in most of the analogous P and As complexes,[3] its distortion is less dramatic than for $[CpMo(\mu,\eta^5\text{-}As_5)MoCp]$,[1] where an As_2+As_3 arrangement was observed (As–As 238.9(2) – 276.2(3)). This is contrary to predictions, based on theoretical calculations, of relatively stronger Jahn – Teller distortions for an Sb_5 ligand in a complex of type **2** compared to its P and As analogues.[14] The pronounced deformation of the arsenic middle deck in $[CpMo(\mu,\eta^5\text{-}As_5)MoCp]$ is likely due to the strong intermolecular interactions involving the As_2 fragments, and not to the Jahn – Teller distortion. In the solid state, the molecules of $[CpMo(\mu,\eta^5\text{-}As_5)MoCp]$ associate to dimers over As ··· As contacts (301, 319, 320, 321 pm; sum of van der Waals radii for arsenic is 400 pm); one of these interactions is the shortest intermolecular As ··· As contact known to date. The two molecules of a dimer lie with the pseudo-C_5 axes perpendicular to each other, the As_2 units building a distorted As_4 tetrahedron. Long intermolecular Sb ··· Sb contacts[15] (397.2, 404.9, 411.0, and 432.6 pm; sum of van der Waals radii for antimony is 440 pm) are responsible for undulating chains of molecules throughout the lattice of **2** and probably determine the slight distortion of the Sb_5 ring.

The Mo(1)–Mo(2) distance (296.40(8) pm) in **2** is considerably longer than that in $[CpMo(\mu,\eta^5\text{-}As_5)MoCp]$ (276.4(2) pm) and corresponds to that of a Mo–Mo single bond.[1] The Sb–Mo bond lengths range between 272.76(8) and 290.23(8) pm, and are comparable to other Sb–Mo bond lengths in complexes with substituent-free antimony ligands: 280.8 pm (av) in $[\{CpMo(CO)_2\}_2Sb_2]$,[6a] 287.88 pm (av) in $[Cp^*Mo(CO)_2Sb_3]$,[8a] and 290.06 pm (av) in $[CpMo(CO)_2Sb_3]$.[8a] An indication for the steric strain in the molecule is the strong bending (12.5 – 17.4°) of the *tert*-butyl groups outside the Cp plane. Both cyclopentadienyl rings are twisted (14.7 and −9.1°) around the Mo(1)–Mo(2) axis with respect to the Sb_5 ring.

Complex **2** (and very likely, **1**) has 27 valence electrons ($3 \times 5 + 2 \times 6$, cf. P and As analogues).[2b, 3] The broadening of the signals in the ^1H NMR spectrum of **1** and **2**, as well as the absence of signals in the ^{13}C NMR spectrum are in agreement with the clusters being paramagnetic.

The observations on the stability of complexes with Sb_n ligands[8] suggest that, in contrast to the P and As analogues, an efficient steric protection is a condition for the isolation of triple-decker sandwich complexes containing the *cyclo*-Sb_5 ligand. Although such clusters possess a remarkable thermal stability in the gas phase, without efficient steric protection by bulky cyclopentadienyl groups they are likely to suffer decomposition in a condensed phase, with formation of metallic antimony. The same trend is shown by the molecules P_4, As_4, and Sb_4. It is to be seen if appropriate steric protection will enable the isolation of a cluster containing a *cyclo*-Sb_6 ligand, and if bismuth can be involved in a similar chemistry.

Experimental Section

1 – 3: All operations were carried out with careful exclusion of air in an argon atmosphere or in vacuo. *cyclo*-tBu_4Sb_4 (1.88 g, 2.63 mmol) and $[Cp'''(CO)_3MoCH_3]$ (1 g, 2.33 mmol) were stirred under reflux in decaline (60 mL) for about 3 h. The solvent was removed in vacuo and the residue extracted with hexane (3 × 100 mL). The green-yellow extracts were combined, Al_2O_3 (8 g) was added and the solvent was removed under reduced pressure. The residue was placed on a chromatography column (3 × 20 cm Al_2O_3 neutral, particle size 0.063 – 0.200 mm, degree of activity II/Brockmann). An emerald-green fraction was eluted with hexane/toluene (99/1). Removal of the solvent in vacuo and recrystallization of the residue from hexane at −30 °C gave 265 mg of a microcrystalline green solid containing both **1** and **2**. Dissolution in toluene followed by slow evaporation in vacuo produced a mixture of a microcrystalline powder with larger crystals, which were selected for X-ray crystallography and shown to be **2**. **1**+**2**: M.p. 331 – 333 °C; elemental analysis (%): calcd for $C_{34}H_{58}Mo_2Sb_5$, $C_{31}H_{52}Mo_2Sb_5$: C 32.22, 30.39, H 4.61, 4.29; found: C 32.18, H 4.63; ^1H NMR (250 MHz, C_6D_6, 25 °C): $\delta = 0.91$ (br.s), 1.35 (br.s), 1.38 (br.s), 3.39 (br.s), 3.58 (br.s), 3.62 (br.s), 3.77 (br.s), 29.04 (br.s); MS (EI, 70 eV): m/z (%): 1266 (100) [**1**, M^+], 1224 (25) [**2**, M^+], 1195 (20) [**1**, $M^+ - C_5H_{11}$], 1153 (10) [**2**, $M^+ - C_5H_{11}$], 1122 (20), 57 (80) [tBu^+]. With hexane/toluene (9/1) was eluted a yellow-brown fraction which left behind, after removal of the solvent, **3** as a dark brown solid. Crystals were obtained by cooling a solution in hexane to −30 °C. Yield: 205 mg (22.8 %). **3**: M.p. 185 – 190 °C; elemental analysis (%): calcd for $C_{38}H_{58}Mo_2O_4$: C 59.22, H 7.58; found: C 60.48, H 7.64; ^1H NMR (250 MHz, C_6D_6, 25 °C): $\delta = 1.25$ (s, 9 H; *t*Bu), 1.43 (s, 18 H; *t*Bu), 5.23 (s, 2 H; C_5H_2); ^{13}C NMR (63 MHz, C_6D_6, 25 °C): $\delta = 31.4, 32.1, 33.8, 34.3$ (*t*Bu), 96.4, 118.3, 123.2 (C_5H_2), 242.4 (CO); IR (Nujol): $\tilde{\nu} = 1943, 1878, 1819, 1802\ cm^{-1}$ (C=O); MS (EI, 70 eV): m/z (%): 770 (80) [M^+], 742 (60) [$M^+ - CO$], 714 (40) [$M^+ - 2CO$], 686 (30) [$M^+ - 3CO$], 650 (43), 57 (100) [tBu^+].

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[10] X-ray crystal structure analysis of **2**: Bruker P4/RA/SMART 1000 CCD diffractometer, Mo$_{K\alpha}$ radiation, $\lambda = 71.073$ pm, scan mode ϕ/ω, $T = 193(2)$ K, absorption correction SADABS, method of structure solution: direct methods, program used for structure solution SHELXS-97,[16] method of refinement full-matrix least-squares at F^2, refinement program SHELXL-97, hydrogen atoms geometrically positioned and refined with a riding model. Crystal dimensions $0.11 \times 0.17 \times 0.22$ mm^3, crystal system monoclinic, space group $P2/n$, $a = 1320.30(6)$, $b = 1002.10(5)$, $c = 2834.96(12)$ pm, $\beta = 95.429(1)^\circ$, $V = 3.7340(3)$ nm^3, $Z = 4$, $\rho_{\text{calcd}} = 2.180$ Mg m^{-3}, $\Theta_{\text{max}} = 26.40^\circ$, 18091 measured reflections, 7653 independent intensities ($R_{\text{int}} = 0.0343$), absorption coefficient 4.230 mm^{-1}, number of free parameters 372, final R ($I > 2\sigma(I)$), $R1 = 0.0474$, $wR2 = 0.0916$. Crystallographic data (excluding structure factors) for the structure reported in this paper have been deposited with the Cambridge Crystallographic Data Centre as supplementary publication no. CCDC-146815. Copies of the data can be obtained free of charge on application to CCDC, 12 Union Road, Cambridge CB21EZ, UK (fax: (+44) 1223-336-033; e-mail: deposit@ccdc.cam.ac.uk).

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Efficient Chemical Synthesis of the Two Anomers of ADP-L-*glycero*- and D-*glycero*-D-*manno*-Heptopyranose Allows the Determination of the Substrate Specificities of Bacterial Heptosyltransferases**

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Lipopolysaccharides (LPS) of gram-negative bacteria are essential outer membrane components. The lipid moiety and the adjacent first sugar residues, named lipid A and the inner core region, respectively, are of utmost importance for growth and viability.[1, 2] Thus, the development of inhibitors of the early biosynthetic steps in the assembly of LPS are promising alternatives in antibacterial drug design.

Whereas the biosynthesis of the 3-deoxy-D-*manno*-oct-2-ulosonic acid (Kdo) and the lipid A region of LPS is well understood, that of the heptose region containing L-*glycero*- or D-*glycero*-D-*manno*-heptose, or both, is not known in detail. This concerns the biosynthesis of the sugars, their nucleotide derivatives, and the characterization of heptosyltransferases.[3–5] Although it is known that some bacterial heptosyltransferases use ADP-heptose as a substrate, the configuration of the side chain and the anomeric carbon is not known for the physiological sugar nucleotide.[3, 6–8]

For the synthesis of the glycopyranosyl 1-phosphates,[9] L-*glycero*-D-*manno*-heptopyranose (**5**)[10] was subjected to phosphitylation[11] followed by oxidation to give the anomeric phosphate triesters **6** and **7** in approximately 9:1 ratio and in 94% yield (Scheme 1). Alternatively, treatment of **5** with *N*-tetrazolylbisbenzylphosphoramidite increased the formation of the β-anomer **7**.[12] By slow addition of phosphitylating reagent in the presence of an excess of 4-dimethylaminopyridine (DMAP), the thermodynamically and kinetically controlled α- and β-anomeric phosphite triesters, respectively, were obtained. Subsequent in situ oxidation gave **6** (56%) and **7** (34%), respectively. Cleavage of the protecting groups by hydrogenolysis to furnish **8** and **12** followed by deacetylation gave α-and β-heptose 1-phosphates (**9** and **13**, respectively) in near quantitative yields.

Yields reported for chemical syntheses of sugar nucleoside diphosphates through the coupling of a sugar 1-phosphate with nucleoside monophosphoromorpholidate[13] are mostly moderate, not exceeding 25–30%, although improvements have been published.[14] Target compound **1** was prepared first

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